



# ***STIC Search Report*** **EIC 1700**

**STIC Database Tracking Number: 175384**

**TO: Ben Sackey**  
**Location: REM 5B31**  
**Art Unit : 1626**  
**January 10, 2006**

**Case Serial Number: 10/719856**

**From: Kathleen Fuller**  
**Location: EIC 1700**  
**REMSEN 4B28**  
**Phone: 571/272-2505**  
**Kathleen.Fuller@uspto.gov**

## **Search Notes**

I think the applicants were blowing smoke with their nomenclature for the compound in claim 1- DNTDFD or that dinosyl part of the name. I finally figured out what the structure was –see the attachments. There were only 3 references in Casreact and 2 in CA.

Protonitronium. See, *Synthesis and Structure of HNF<sub>x</sub>*, Robert D. Chapman, Journal of Organic Chemistry (1999, 64, 960-965).

The known methods for producing DNTDFD are described in Disfluoramination of Heterocyclic Ketones: Control of Microbasicity, Robert D. Chapman, Journal of Organic Chemistry (1998, 63, 1566). The compound HNF<sub>x</sub> is similar to another explosive and propellant oxidizer, TNFX. The process of preparing TNFX is detailed in U.S. Patent Application No. 2000/0161248, to Chapman and in U.S. Patent No. 6,417,355, also to Chapman. The precursor to TNFX is a compound very similar to DNTDFD. Both compounds are synthesized by a lengthy process requiring fluctuating temperature conditions. In preparing the precursor to TNFX, the reaction proceeds slowly and must be encouraged by cycling the temperature between -15 degrees C and 0 degrees C throughout the process, which reaches completion in two weeks time.

The process of preparing DNTDFD, as described in *Disfluoramination of Heterocyclic Ketones: Control of Microbasicity*, Robert D. Chapman, Journal of Organic Chemistry (1998, 63, 1566 at 1570), requires a solution of fuming sulfuric acid, to which CH<sub>2</sub>Cl<sub>2</sub> is added and cooled to -15 degrees C. Gaseous HNF<sub>2</sub> is absorbed into the layer of CH<sub>2</sub>Cl<sub>2</sub>. The temperature is raised, briefly, so that the HNF<sub>2</sub> may be absorbed and the mixture is recooled. Tetrahydro-1,5-bis(4-nitrobenzenesulfonyl)-1,5-diazocine-3,7-(2H, 6H) dione is added to the mixture, which is stirred for 15 days. The solution is basified and precipitated. The product, DNTDFD, is obtained. During the course of the reaction, temperature is allowed to rise gradually from -15 degrees C to -8 degrees C. An alternate method is described, which involves the absorption of HNF<sub>2</sub> gas into a layer of FREON<sup>®</sup> 11 and the addition of tetrahydro - 1, 5-bis(4 -

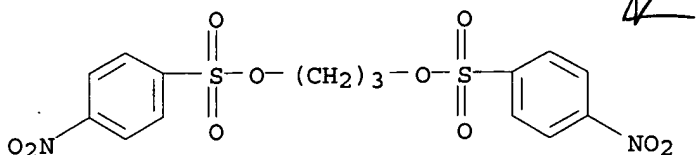
L6 ANSWER 1 OF 2 REGISTRY COPYRIGHT 2006 ACS on STN  
RN 25297-83-0 REGISTRY  
ED Entered STN: 16 Nov 1984  
CN Benzenesulfonic acid, 4-nitro-, 1,3-propanediyl ester (9CI) (CA INDEX NAME)

## OTHER CA INDEX NAMES:

CN 1,3-Propanediol, bis(p-nitrobenzenesulfonate) (8CI)  
CN Benzenesulfonic acid, p-nitro-, trimethylene ester (8CI)

## OTHER NAMES:

CN **1,3-Propylene glycol dinosylate**  
FS 3D CONCORD  
MF C15 H14 N2 O10 S2  
LC STN Files: CA, CAPLUS, TOXCENTER



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

3 REFERENCES IN FILE CA (1907 TO DATE)  
3 REFERENCES IN FILE CAPLUS (1907 TO DATE)

=> D 2

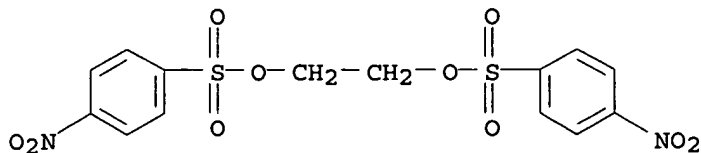
L6 ANSWER 2 OF 2 REGISTRY COPYRIGHT 2006 ACS on STN  
RN 25297-82-9 REGISTRY  
ED Entered STN: 16 Nov 1984  
CN Benzenesulfonic acid, 4-nitro-, 1,2-ethanediyl ester (9CI) (CA INDEX NAME)

## OTHER CA INDEX NAMES:

CN Benzenesulfonic acid, p-nitro-, ethylene ester (8CI)  
CN Ethylene glycol, bis(p-nitrobenzenesulfonate) (8CI)

## OTHER NAMES:

CN **Ethylene glycol dinosylate**  
CN NSC 115800  
FS 3D CONCORD  
MF C14 H12 N2 O10 S2  
LC STN Files: CA, CAPLUS, TOXCENTER



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

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Scientific and Technical Information Center

SEARCH REQUEST FORM

Requester Full Name: GEN SACKLEY Examiner #: 73489 Date: 12/30/05  
Art Unit: 1626 Phone Number: 6204 Serial Number: 101719, 856  
Location (Bldg/Room#): REM 5631 Mailbox #: \_\_\_\_\_ Results Format Preferred (circle): PAPER DISK

To ensure an efficient and quality search, please attach a copy of the cover sheet, claims, and abstract or fill out the following:

Title of Invention: Process for Synthesizing 1,5-dioxol-3,3,7,7-tetrakis(difluoraminio) octahydro-1,5-diazocine (DATD7D)  
Inventors (please provide full names): Adolph et al.

Earliest Priority Date: 11/24/03

Search Topic:

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known.

\*For Sequence Searches: Please include all relevant information (sequence, date, divisional, or issued patent numbers) along with the appropriate serial number.

4. process for prep. 1,5-dioxol-3,3,7,7-tetrakis(difluoraminio) octahydro-1,5-diazocine (DATD7D) comprising reacting tetrahydro-1,5-bis(4-nitrobenzene sulfonyl)-4,5-diazocine-3,7-(2H,6H) dione with HNF<sub>2</sub>/oleum in the presence of a diluent and absorbent for the reagent difluoramine.

Note: diluent -> mixture of toluene with pentane, or cyclopentane or pentane alone

Thanks

STAFF USE ONLY

Searcher: K. Fuller

Searcher Phone #: \_\_\_\_\_

Searcher Location: \_\_\_\_\_

Date Searched: \_\_\_\_\_

Date Constructed: 1/10/06

Searcher Prep & Review Time: 40

Online Time: 60

Type of Search

NA Sequence ID

AA Sequence ID

2 Sequence ID

Sequence ID

Literature

Abstract

Other

Vendors and cost where applicable

☒ STN ☐ Dialog

☐ Questel/Orbit ☐ Lexis/Nexis

☐ Westlaw ☐ WWW/Internet

☐ In-house sequence systems

☐ Commercial ☐ Oligomer ☐ Score/Length

☐ Interference ☐ SPDI ☐ Encoder/Transl

Other (specify): \_\_\_\_\_

=> FILE REG

FILE 'REGISTRY' ENTERED AT 13:39:20 ON 10 JAN 2006

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STRUCTURE FILE UPDATES: 9 JAN 2006 HIGHEST RN 871542-42-6

DICTIONARY FILE UPDATES: 9 JAN 2006 HIGHEST RN 871542-42-6

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TSCA INFORMATION NOW CURRENT THROUGH JULY 14, 2005

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\*\*\*\*\*  
\*  
\* The CA roles and document type information have been removed from \*  
\* the IDE default display format and the ED field has been added, \*  
\* effective March 20, 2005. A new display format, IDERL, is now \*  
\* available and contains the CA role and document type information. \*  
\*  
\*\*\*\*\*

Structure search iteration limits have been increased. See HELP SLIMITS for details.

REGISTRY includes numerically searchable data for experimental and predicted properties as well as tags indicating availability of experimental property data in the original document. For information on property searching in REGISTRY, refer to:

<http://www.cas.org/ONLINE/UG/regprops.html>

=> D L9

L9 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2006 ACS on STN

RN 202211-20-9 REGISTRY

ED Entered STN: 05 Mar 1998

CN 1,5-Diazocine-3,3,7,7(2H,4H)-tetramine, N,N,N',N',N'',N'',N''',N''''-  
octafluorotetrahydro-1,5-bis[(4-nitrophenyl)sulfonyl]- (9CI) (CA INDEX  
NAME)

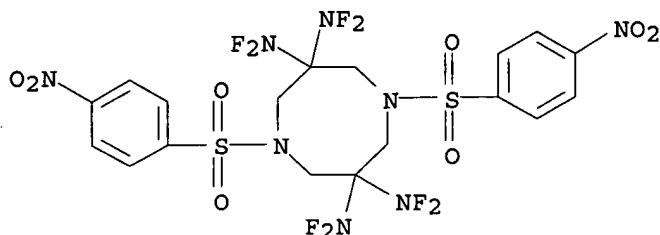
FS 3D CONCORD

MF C18 H16 F8 N8 O8 S2

SR CA

LC STN Files: CA, CAPLUS, CASREACT

*This is the  
desired compound*



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

2 REFERENCES IN FILE CA (1907 TO DATE)

2 REFERENCES IN FILE CAPLUS (1907 TO DATE)

*only 2 references*

=> FILE HCAPLU

FILE 'HCAPLUS' ENTERED AT 13:39:40 ON 10 JAN 2006

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FILE COVERS 1907 - 10 Jan 2006 VOL 144 ISS 3

FILE LAST UPDATED: 9 Jan 2006 (20060109/ED)

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This file contains CAS Registry Numbers for easy and accurate substance identification.

=> D QUE

L9 1 SEA FILE=REGISTRY ABB=ON 202211-20-9

L10 2 SEA FILE=HCAPLUS ABB=ON L9

=> D L10 1-2 ALL

L10 ANSWER 1 OF 2 HCAPLUS COPYRIGHT 2006 ACS on STN

AN 1999:30927 HCAPLUS

DN 130:209682

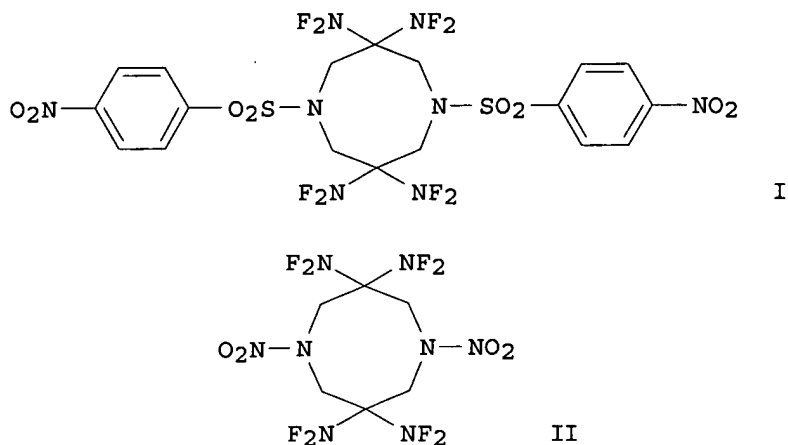
ED Entered STN: 18 Jan 1999

TI Nitrolysis of a highly deactivated amide by protonitronium. Synthesis and structure of HNFx

AU Chapman, Robert D.; Gilardi, Richard D.; Welker, Mark F.; Kreutzberger, Charles B.

CS Naval Aviation Science Technology Office, Naval Air Warfare Center Weapons

Division, China Lake, CA, 93555, USA  
SO Journal of Organic Chemistry (1999), 64(3), 960-965  
CODEN: JOCEAH; ISSN: 0022-3263  
PB American Chemical Society  
DT Journal  
LA English  
CC 28-23 (Heterocyclic Compounds (More Than One Hetero Atom))  
Section cross-reference(s): 50, 75  
OS CASREACT 130:209682  
GI



AB Efficient N-nitrolysis of the highly deactivated  
tetrakis(difluoramino)octahydrobis(4-nitrobenzenesulfonyl)diazocine I was  
achieved by the use of a protonitronium reagent formed in the system  
nitric acid-trifluoromethanesulfonic acid, producing  
tetrakis(difluoramino)octahydrodinitrodiazocine (II; HNFX) in 65% yield in  
a nonoptimized reaction. The crystal structure of the first morphol. of  
II contains cavities in the form of channels through its unit cell.

ST fluoroaminodiazocine bisnitrobenzenesulfonyl nitrolysis; nitrolysis  
protonitronium reagent nitrobenzenesulfonyldifluoroaminodiazocine;  
diazocine dinitrotetrakis(difluoroamino) prepn mol crystal structure; HNFX  
prepn mol structure

IT Crystal structure  
Molecular structure  
(of tetrakis(difluoroamino)dinitrodiazocine)

IT Nitration  
(preparation of tetrakis(difluoroamino)dinitrodiazocine by nitrolysis of  
bis(nitrophenylsulfonyl)diazocine derivative)

IT 170787-71-0P, HNFX  
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
(preparation and mol./crystal structure of tetrakis(difluoroamino)dinitrodia  
zocine)

IT 202211-14-1 202211-20-9  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(preparation and mol./crystal structure of tetrakis(difluoroamino)dinitrodia  
zocine)

IT 220841-48-5P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT

(Reactant or reagent)

(preparation and mol./crystal structure of tetrakis(difluoroamino)dinitrodiazocine)

RE.CNT 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD  
RE

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L10 ANSWER 2 OF 2 HCAPLUS COPYRIGHT 2006 ACS on STN

AN 1998:157886 HCAPLUS

DN 128:140683

ED Entered STN: 17 Mar 1998

TI Difluoramination of Heterocyclic Ketones: Control of Microbasicity

AU Chapman, Robert D.; Welker, Mark F.; Kreutzberger, Charles B.

CS Research and Technology Group (Code 4B2200D), Naval Air Warfare Center  
Weapons Division, China Lake, CA, 93555, USA

SO Journal of Organic Chemistry (1998), 63(5), 1566-1570

CODEN: JOCEAH; ISSN: 0022-3263

PB American Chemical Society

DT Journal

LA English

CC 28-23 (Heterocyclic Compounds (More Than One Hetero Atom))

Section cross-reference(s): 50

OS CASREACT 128:140683

AB Difluoramination of a tetrahydro-1,5-diazocine-3,7(2H,6H)-dione to the  
corresponding 3,3,7,7-tetrakis(difluoramino)diazocine was achieved by a  
judicious choice of protecting group. Arenesulfonyl protecting groups for

*This reference is  
cited in the specifications  
p2 as a prep for DNTDFB  
though it does not  
call it that.*



the diazocine nitrogens proved superior to acetyl during the slow disruption of the transannular bridge in 9-oxa-3,7-diazabicyclo[3.3.1]nonane intermediates by difluorosulfamic acid. While a 1,5-ditosyl derivative failed to proceed beyond the product of addition of difluoramine to one ketone carbonyl, the use of 4-nitrobenzenesulfonyl as the protecting groups lowered the nitrogens' basicities below that of the oxygen site in the dione and intermediates, allowing the reaction to proceed to a gem-bis(difluoramino)diazocine product. A safer procedure for handling difluoramine is described.

- ST tetrahydrodiazocinedione difluoramination; diazocinedione tetrahydro difluoramination; difluoramination heterocyclic ketone; safety handling difluoramine
- IT Amination  
(difluoramination of tetrahydrodiazocinedione)
- IT Protective groups  
(effect of protecting groups in difluoramination of tetrahydrodiazocinedione)
- IT Safety  
(in handling difluoramine)
- IT 106-89-8, reactions 1510-31-2 6325-93-5 94683-14-4  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(difluoramination of tetrahydrodiazocinedione)
- IT 10405-27-3P, Difluoramine 160624-80-6P 202211-14-1P 202211-16-3P  
202211-17-4P 202211-18-5P 202211-19-6P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(difluoramination of tetrahydrodiazocinedione)
- IT 202211-15-2P **202211-20-9P**  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(difluoramination of tetrahydrodiazocinedione)

RE.CNT 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

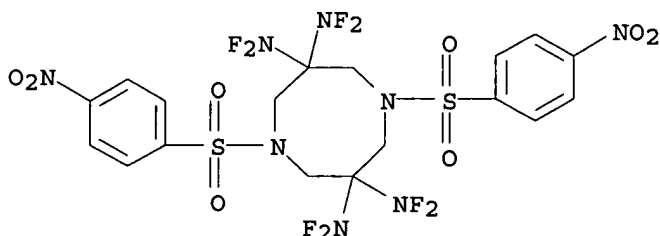
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- (2) Baum; Final report to the Office of Naval Research (Arlington, VA) on Contract N00014-88-C-0536 1991, ONR-7-1
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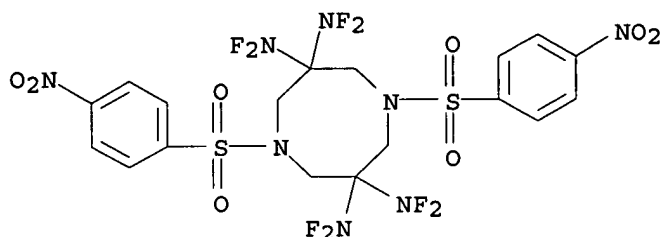
=> D L10 TI HITSTR 1-2

*structures for above 2 references*

L10 ANSWER 1 OF 2 HCAPLUS COPYRIGHT 2006 ACS on STN  
TI Nitrolysis of a highly deactivated amide by protonitronium. Synthesis and structure of HNFX  
IT 202211-20-9  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(preparation and mol./crystal structure of tetrakis(difluoroamino)dinitrodiazocine)  
RN 202211-20-9 HCAPLUS  
CN 1,5-Diazocine-3,3,7,7(2H,4H)-tetramine, N,N,N',N',N'',N'',N''',N'''-octafluorotetrahydro-1,5-bis[(4-nitrophenyl)sulfonyl]- (9CI) (CA INDEX NAME)



L10 ANSWER 2 OF 2 HCAPLUS COPYRIGHT 2006 ACS on STN  
TI Difluoramination of Heterocyclic Ketones: Control of Microbasicity  
IT 202211-20-9P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(difluoramination of tetrahydrodiazocinedione)  
RN 202211-20-9 HCAPLUS  
CN 1,5-Diazocine-3,3,7,7(2H,4H)-tetramine, N,N,N',N',N'',N'',N''',N'''-octafluorotetrahydro-1,5-bis[(4-nitrophenyl)sulfonyl]- (9CI) (CA INDEX NAME)



=> => FILE CASREAC

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FILE CONTENT:1840 - 8 Jan 2006 VOL 144 ISS 2

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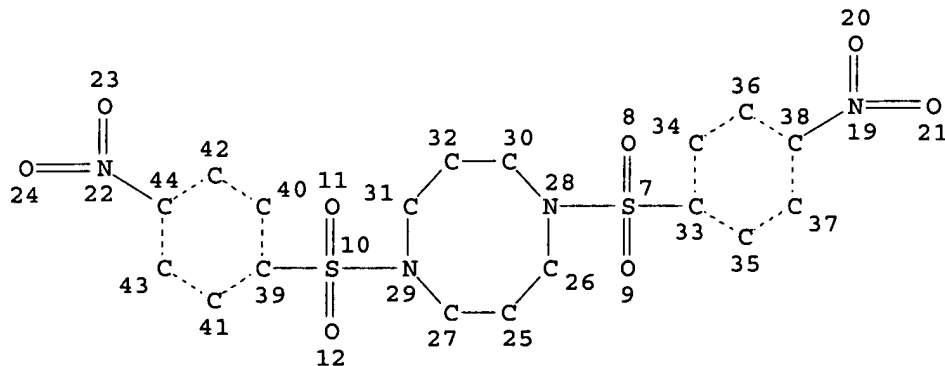
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 \* CASREACT now has more than 10 million reactions \*  
 \*  
 \*\*\*\*\*

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This file contains CAS Registry Numbers for easy and accurate substance identification.

=> D QUE

L11 STR



*any reaction  
with this*

NODE ATTRIBUTES:  
 DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

## GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS 32

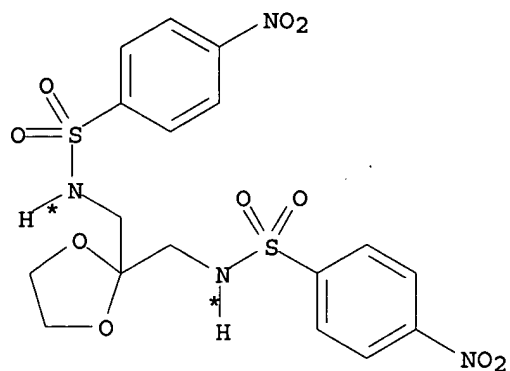
## STEREO ATTRIBUTES: NONE

L13 3 SEA FILE=CASREACT SSS FUL L11 ( 52 REACTIONS)

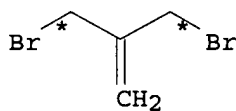
=&gt; D L13 FHIT BIB ABS IND

L13 ANSWER 1 OF 3 CASREACT COPYRIGHT 2006 ACS on STN

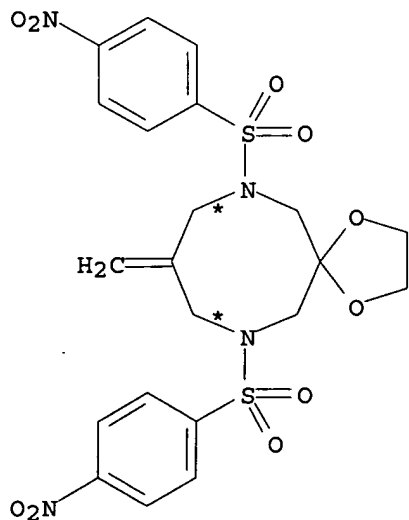
RX(4) OF 100 ...J + M ==&gt; N...



J



M



N

YIELD 76%

RX(4) RCT J 333421-17-3, M 15378-31-1  
 RGT O 584-08-7 K2CO3  
 PRO N 333421-21-9  
 SOL 67-64-1 Me2CO

AN 137:78873 CASREACT

TI Preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX), related intermediates and use in explosives and propellant oxidizers

IN Chapman, Robert Dale; Axenrod, Theodore; Sun, Jianguang; Guan, Xiao-Pei; Qi, Lida

PA United States Dept. of the Navy, USA

SO U.S., 12 pp.

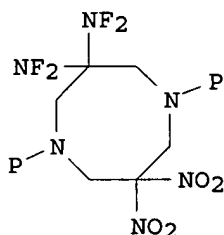
CODEN: USXXAM

DT Patent

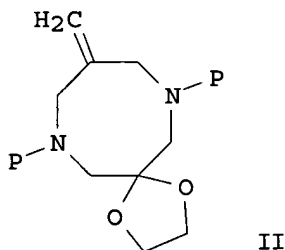
LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 6417355	B1	20020709	US 2001-835783	20010411
	US 2002161248	A1	20021031	US 2002-166278	20020603
	US 6562985	B2	20030513		
PRAI	US 2001-835783		20010411		
OS	MARPAT 137:78873				
GI					



I



II

AB Disclosed is a process for the preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine [I; P = NO2]. Intermediate II [prepared in 4 steps; P = 4-nitrobenzenesulfonyl] subjected to the following steps:  
 i. CH2Cl2, O3, -78°C/Me2S; ii. EtOH, NH2OH•HCl, NaOAc, reflux;  
 iii. CH2Cl2, HNO3/NH4NO3/urea, reflux; iv. CH2Cl2, H2SO4 and v. CFCl3, H2SO4, HNF2, -25° → 10° → -15°, 3 h to produce, after aqueous work-up, the acetone solvate of I (explosive; P = 4-nitrobenzenesulfonyl; III). III was converted to I by treatment with CF3SO3H, HNO3, at 55° and aging the mixture for 14 days followed by addition of SbF5. Removal of triflic acid by distillation followed by aqueous work-up yielded the title compound I provides a difluoroamino component desired for energetic combustion of metalized-fuel propellant formulations, and the gem-dinitro component provides higher oxygen balance (for more-complete combustion) than analogous all-difluoroamino derivs.

IC ICM C07D225-04

ICS C07D245-00

NCL 540466000

CC 27-21 (Heterocyclic Compounds (One Hetero Atom))

Section cross-reference(s): 50

ST safety geminal dinitro diazocine explosion difluoroamino propellant combustion

IT Combustion

Explosion  
Explosives  
Nitration  
Propellants (fuels)  
    (preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX), related intermediates and use in explosives and propellant oxidizers)

IT Lewis acids  
RL: RGT (Reagent); RACT (Reactant or reagent)  
    (preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX), related intermediates and use in explosives and propellant oxidizers)

IT Nitramines  
RL: SPN (Synthetic preparation); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)  
    (preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX), related intermediates and use in explosives and propellant oxidizers)

IT 193021-35-1P  
RL: SPN (Synthetic preparation); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)  
    (combustible, explosive; preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX), related intermediates and use in explosives and propellant oxidizers)

IT 440651-48-9P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
    (intermediate, explodes!; preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX) and related intermediates)

IT 333421-07-1P 333421-09-3P 333421-11-7P 333421-13-9P 333421-15-1P  
333421-17-3P 333421-19-5P 333421-21-9P 333421-23-1P 333421-25-3P  
333421-27-5P 333421-29-7P 333421-31-1P 333421-33-3P 333421-35-5P  
333421-36-6P 333421-37-7P 440651-52-5P 440651-53-6P,  
Hexahydro-7,7-dinitro-1,5-bis(3-fluorobenzenesulfonyl)-1,5-diazocin-3(2H)-one 440651-54-7P, Hexahydro-7,7-dinitro-1,5-bis(4-fluorobenzenesulfonyl)-1,5-diazocin-3(2H)-one 440651-55-8P, Hexahydro-7,7-dinitro-1,5-bis(2-cyanobenzenesulfonyl)-1,5-diazocin-3(2H)-one 440651-56-9P,  
Hexahydro-7,7-dinitro-1,5-bis(3-cyanobenzenesulfonyl)-1,5-diazocin-3(2H)-one 440651-57-0P, Hexahydro-7,7-dinitro-1,5-bis(4-cyanobenzenesulfonyl)-1,5-diazocin-3(2H)-one 440651-58-1P, Hexahydro-7,7-dinitro-1,5-bis(3-nitrobenzenesulfonyl)-1,5-diazocin-3(2H)-one 440651-59-2P,  
3,3-Bis(difluoroamino)octahydro-7,7-di(nitro)-1,5-bis(2-fluorobenzenesulfonyl)-1,5-diazocine 440651-60-5P, 3,3-Bis(difluoroamino)octahydro-7,7-di(nitro)-1,5-bis(3-fluorobenzenesulfonyl)-1,5-diazocine 440651-61-6P, 3,3-Bis(difluoroamino)octahydro-7,7-di(nitro)-1,5-bis(4-fluorobenzenesulfonyl)-1,5-diazocine 440651-62-7P,  
3,3-Bis(difluoroamino)octahydro-7,7-di(nitro)-1,5-bis(2-cyanobenzenesulfonyl)-1,5-diazocine 440651-63-8P, 3,3-Bis(difluoroamino)octahydro-7,7-di(nitro)-1,5-bis(3-cyanobenzenesulfonyl)-1,5-diazocine 440651-64-9P, 3,3-Bis(difluoroamino)octahydro-7,7-di(nitro)-1,5-bis(4-cyanobenzenesulfonyl)-1,5-diazocine 440651-65-0P,  
3,3-Bis(difluoroamino)octahydro-7,7-di(nitro)-1,5-bis(3-nitrobenzenesulfonyl)-1,5-diazocine 440651-66-1P 440651-67-2P  
440651-68-3P 440651-69-4P 440651-70-7P 440651-71-8P 440651-72-9P  
440651-73-0P 440651-74-1P 440651-75-2P 440651-76-3P 440651-77-4P  
440651-78-5P 440651-79-6P 440651-80-9P 440651-81-0P 440651-82-1P  
440651-83-2P 440651-84-3P 440651-85-4P 440651-86-5P 440651-87-6P  
440651-88-7P 440651-89-8P 440651-90-1P 440651-91-2P 440651-92-3P  
440651-93-4P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT

(Reactant or reagent)  
(intermediate; preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX) and related intermediates)

IT 333421-34-4P, Hexahydro-7,7-di(nitro)-1,5-bis(2-nitrobenzenesulfonyl)-1,5-diazocin-3(2H)-one  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(intermediate; preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX), related intermediates and use in explosives and propellant oxidizers)

IT 7783-70-2, Antimony pentafluoride 64371-01-3, Boron triflate 440651-94-5  
RL: RGT (Reagent); RACT (Reactant or reagent)  
(nitration catalyst; preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX) and related intermediates)

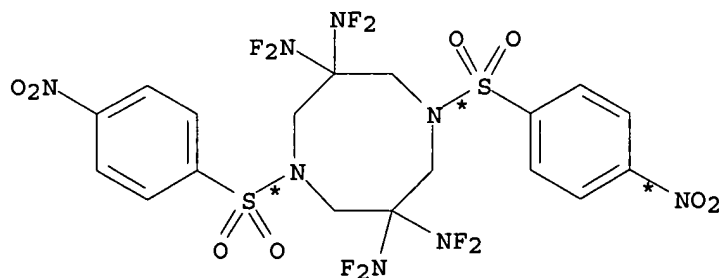
IT 98-74-8, p-Nitrobenzenesulfonyl chloride 616-29-5 1694-92-4, 2-Nitrobenzenesulfonyl chloride 15378-31-1, 3-Bromo-2-(bromomethyl)propene  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(reactant; preparation of 3,3-bis(difluoroamino)octahydro-1,5,7,7-tetra(nitro)-1,5-diazocine (TNFX) and related intermediates)

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> D L13 FHIT BIB ABS IND 2-3

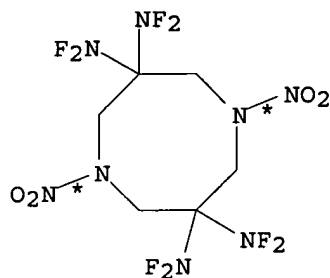
L13 ANSWER 2 OF 3 CASREACT COPYRIGHT 2006 ACS on STN

RX(1) OF 1 A ==> B



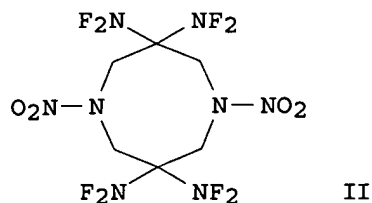
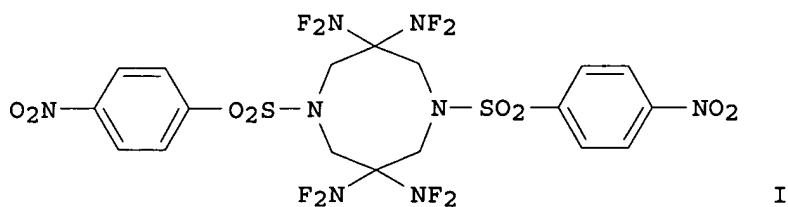
A

(1) →



B  
YIELD 65%

RX(1) RCT A 202211-20-9  
 RGT C 7697-37-2 HNO3, D 1493-13-6 F3CSO2H  
 PRO B 170787-71-0  
 AN 130:209682 CASREACT  
 TI Nitrolysis of a highly deactivated amide by protonitronium. Synthesis and structure of HNFX  
 AU Chapman, Robert D.; Gilardi, Richard D.; Welker, Mark F.; Kreutzberger, Charles B.  
 CS Naval Aviation Science Technology Office, Naval Air Warfare Center Weapons Division, China Lake, CA, 93555, USA  
 SO Journal of Organic Chemistry (1999), 64(3), 960-965  
 CODEN: JOCEAH; ISSN: 0022-3263  
 PB American Chemical Society  
 DT Journal  
 LA English  
 GI



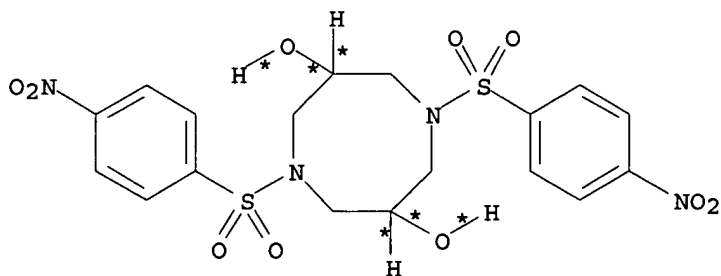
AB Efficient N-nitrolysis of the highly deactivated tetrakis(difluoramino)octahydrobis(4-nitrobenzenesulfonyl)diazocine I was achieved by the use of a protonitronium reagent formed in the system nitric acid-trifluoromethanesulfonic acid, producing tetrakis(difluoramino)octahydrodinitrodiazocine (II; HNFX) in 65% yield in a nonoptimized reaction. The crystal structure of the first morphol. of



II contains cavities in the form of channels through its unit cell.  
CC 28-23 (Heterocyclic Compounds (More Than One Hetero Atom))  
Section cross-reference(s): 50, 75  
ST fluoroaminodiazocine bisnitrobenzenesulfonyl nitrolysis; nitrolysis  
protonitronium reagent nitrobenzenesulfonyldifluoroaminodiazocine;  
diazocine dinitrotetrakisdifluoroamino prepn mol crystal structure; HNFx  
prepn mol structure  
IT Crystal structure  
Molecular structure  
(of tetrakis(difluoroamino)dinitrodiazocine)  
IT Nitration  
(preparation of tetrakis(difluoroamino)dinitrodiazocine by nitrolysis of  
bis(nitrophenylsulfonyl)diazocine derivative)  
IT 170787-71-0P, HNFx  
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
(preparation and mol./crystal structure of tetrakis(difluoroamino)dinitrodia  
zocine)  
IT 202211-14-1 202211-20-9  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(preparation and mol./crystal structure of tetrakis(difluoroamino)dinitrodia  
zocine)  
IT 220841-48-5P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(preparation and mol./crystal structure of tetrakis(difluoroamino)dinitrodia  
zocine)  
RE.CNT 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

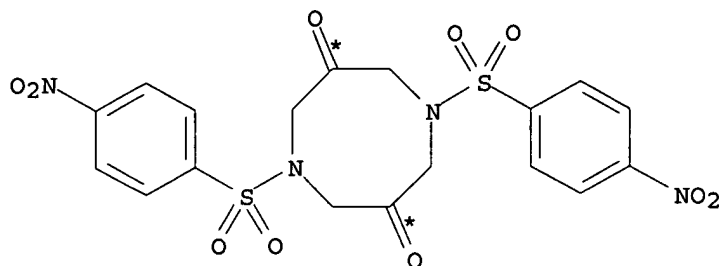
L13 ANSWER 3 OF 3 CASREACT COPYRIGHT 2006 ACS on STN

RX(1) OF 6 ...A ==> B...



A

(1) →



B  
YIELD 94%

RX(1) RCT A 202211-16-3

STAGE(1)

RGT C 67-68-5 DMSO  
SOL 75-09-2 CH2Cl2

STAGE(2)

RGT D 79-37-8 (COCl)<sub>2</sub>  
SOL 75-09-2 CH2Cl2

STAGE(3)

RGT E 121-44-8 Et<sub>3</sub>N

STAGE(4)

RGT F 7732-18-5 Water

PRO B 202211-17-4

NTE Swern oxidn.

AN 128:140683 CASREACT

TI Difluoramination of Heterocyclic Ketones: Control of Microbasicity

AU Chapman, Robert D.; Welker, Mark F.; Kreutzberger, Charles B.

CS Research and Technology Group (Code 4B2200D), Naval Air Warfare Center  
Weapons Division, China Lake, CA, 93555, USA

SO Journal of Organic Chemistry (1998), 63(5), 1566-1570

CODEN: JOCEAH; ISSN: 0022-3263

PB American Chemical Society

DT Journal

LA English

AB Difluoramination of a tetrahydro-1,5-diazocine-3,7(2H,6H)-dione to the corresponding 3,3,7,7-tetrakis(difluoramino)diazocine was achieved by a judicious choice of protecting group. Arenesulfonyl protecting groups for the diazocine nitrogens proved superior to acetyl during the slow disruption of the transannular bridge in 9-oxa-3,7-diazabicyclo[3.3.1]nonane intermediates by difluorosulfamic acid. While a 1,5-ditosyl derivative failed to proceed beyond the product of addition of difluoramine to one ketone carbonyl, the use of 4-nitrobenzenesulfonyl as the protecting groups lowered the nitrogens' basicities below that of the oxygen site in the dione and intermediates, allowing the reaction to proceed to a gem-bis(difluoramino)diazocine product. A safer procedure for handling difluoramine is described.

CC 28-23 (Heterocyclic Compounds (More Than One Hetero Atom))

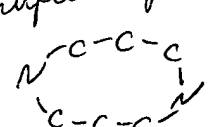
Section cross-reference(s): 50

ST tetrahydrodiazocinedione difluoramination; diazocinedione tetrahydro

difluoramination; difluoramination heterocyclic ketone; safety handling  
difluoramine  
IT Amination  
(difluoramination of tetrahydrodiazocinedione)  
IT Protective groups  
(effect of protecting groups in difluoramination of  
tetrahydrodiazocinedione)  
IT Safety  
(in handling difluoramine)  
IT 106-89-8, reactions 1510-31-2 6325-93-5 94683-14-4  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(difluoramination of tetrahydrodiazocinedione)  
IT 10405-27-3P, Difluoramine 160624-80-6P 202211-14-1P 202211-16-3P  
202211-17-4P 202211-18-5P 202211-19-6P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(difluoramination of tetrahydrodiazocinedione)  
IT 202211-15-2P 202211-20-9P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(difluoramination of tetrahydrodiazocinedione)  
RE.CNT 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> => D QUE

L14 716 SEA FILE=REGISTRY ABB=ON 209.119.1/RID  
L15 364 SEA FILE=REGISTRY ABB=ON L14 AND 46.150.18/RID  
L16 143 SEA FILE=REGISTRY ABB=ON L15 AND 3/NR  
L17 53 SEA FILE=REGISTRY ABB=ON L16 AND 2/S  
L19 1 SEA FILE=REGISTRY ABB=ON FLUORIMIDE/CN  
L20 36 SEA FILE=HCAPLUS ABB=ON L17  
L21 267 SEA FILE=HCAPLUS ABB=ON L19  
L22 2 SEA FILE=HCAPLUS ABB=ON L20 AND L21  
L23 27 SEA FILE=HCAPLUS ABB=ON L20 (L) PREP/RL  
L24 1 SEA FILE=REGISTRY ABB=ON "FREON 11"/CN  
L25 6602 SEA FILE=HCAPLUS ABB=ON L24  
L26 1 SEA FILE=HCAPLUS ABB=ON L25 AND L23  
L27 29 SEA FILE=HCAPLUS ABB=ON L20 AND PREP/RL  
L28 1 SEA FILE=HCAPLUS ABB=ON L27 AND (FREON OR L25)  
L29 2 SEA FILE=HCAPLUS ABB=ON L22 OR L26 OR L28

*ring identified for*  


=> D L29 BIB ABS IND HITSTR 1-2

L29 ANSWER 1 OF 2 HCAPLUS COPYRIGHT 2006 ACS on STN  
AN 2001:222228 HCAPLUS  
DN 134:282903  
TI Synthesis of 3,3-bis(difluoramino)octahydro-1,5,7,7-tetranitro-1,5-  
diazocine (TNFX), a diversified energetic heterocycle  
AU Axenrod, T.; Guan, X.-P.; Sun, J.; Qi, L.; Chapman, R. D.; Gilardi, R. D.  
CS Department of Chemistry, The City College of the City University of New  
York, New York, NY, 100031, USA  
SO Tetrahedron Letters (2001), 42(14), 2621-2623  
CODEN: TELEAY; ISSN: 0040-4039  
PB Elsevier Science Ltd.  
DT Journal  
LA English  
AB The syntheses of new 3,3-dinitro derivs. of the 1,5-diazocine ring system  
are described. Highly deactivated precursor ketones hexahydro-7,7-dinitro-  
1,5-bis(2- and 4-nitrobenzenesulfonyl)-1,5-diazocin-3(2H)-ones were

difluoramined to the corresponding gem-bis(difluoramino)diazocines. The 1,5-bis(4-nitrobenzenesulfonyl)diazocine derivative underwent N-nitrolysis with the protonitronium reagent formed in the nitric acid-trifluoromethanesulfonic acid-antimony pentafluoride system to produce 3,3-bis(difluoramino)octahydro-1,5,7,7-tetranitro-1,5-diazocine (TNFX), containing nitramine, gem-dinitro, and gem-bis(difluoramino) structural components.

CC 50-2 (Propellants and Explosives)  
Section cross-reference(s): 28

ST TNFX fluoramino nitrodiazocine heterocyclic explosive; fluoramination gem nitration heterocycle explosive; nitramine fluoramino diazocine heterocycle explosive

IT Amination  
(difluoroamination; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT Nitration  
(gem-dinitration; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT Oxidation  
Oximation  
(in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT Explosives  
(nitramine-type; synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 75-69-4, Trichlorofluoromethane 7664-93-9, Sulfuric acid, uses 10405-27-3, Difluoroamine 41026-05-5, Sulfamic acid, difluoro-  
RL: NUU (Other use, unclassified); USES (Uses)  
(difluoroamination reagent containing; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 1493-13-6, Trifluoromethanesulfonic acid 7697-37-2, Nitric acid, uses 7783-70-2, Antimony pentafluoride  
RL: NUU (Other use, unclassified); USES (Uses)  
(nitrating reagent containing; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 616-29-5, 1,3-Diamino-2-propanol  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(nitrobenzenesulfonylation of; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 333421-36-6P 333421-37-7P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(synthesis and deprotection-nitration of; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 333421-34-4P 333421-35-5P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(synthesis and difluoroamination of; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 333421-27-5P 333421-29-7P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(synthesis and gem-dinitration of; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 333421-31-1P 333421-33-3P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(synthesis and hydrolysis-deprotection of; in synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 333421-07-1P 333421-09-3P

RL: RCT (Reactant); SPN (Synthetic preparation); **PREP**  
**(Preparation)**; RACT (Reactant or reagent)  
 (synthesis and oxidation of; in synthesis of TNFX,  
 (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 333421-23-1P 333421-25-3P  
 RL: RCT (Reactant); SPN (Synthetic preparation); **PREP**  
**(Preparation)**; RACT (Reactant or reagent)  
 (synthesis and oximation of; in synthesis of TNFX,  
 (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 333421-11-7P 333421-13-9P  
 RL: RCT (Reactant); SPN (Synthetic preparation); **PREP**  
**(Preparation)**; RACT (Reactant or reagent)  
 (synthesis and protection of; in synthesis of TNFX,  
 (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

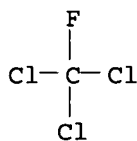
IT 333421-15-1P 333421-17-3P  
 RL: RCT (Reactant); SPN (Synthetic preparation); **PREP**  
**(Preparation)**; RACT (Reactant or reagent)  
 (synthesis and ring closure of; in synthesis of TNFX,  
 (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 193021-35-1P, 1,5-Diazocine-3,3(2H)-diamine, N,N,N',N'-  
 tetrafluoro-hexahydro-1,5,7,7-tetranitro-  
 RL: SPN (Synthetic preparation); **PREP (Preparation)**  
 (synthesis of TNFX, (difluoramino)tetranitrodiazocine, as novel  
 energetic heterocycle)

IT 333421-19-5P 333421-21-9P  
 RL: RCT (Reactant); SPN (Synthetic preparation); **PREP**  
**(Preparation)**; RACT (Reactant or reagent)  
 (synthesis of and ketone formation from; in synthesis of TNFX,  
 (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

IT 75-69-4, Trichlorofluoromethane 10405-27-3,  
 Difluoroamine  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (difluoramination reagent containing; in synthesis of TNFX,  
 (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

RN 75-69-4 HCAPLUS  
 CN Methane, trichlorofluoro- (8CI, 9CI) (CA INDEX NAME)

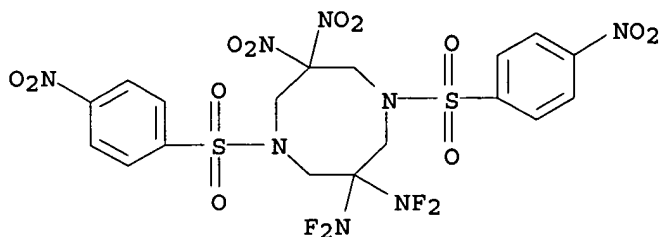


RN 10405-27-3 HCAPLUS  
 CN Fluorimide (6CI, 8CI, 9CI) (CA INDEX NAME)

F-NH-F

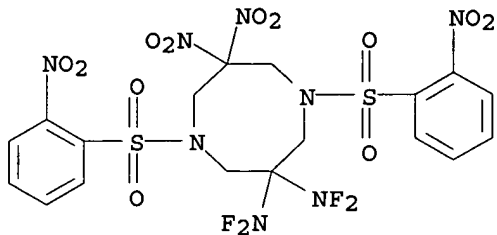
IT 333421-36-6P 333421-37-7P  
 RL: RCT (Reactant); SPN (Synthetic preparation); **PREP**  
**(Preparation)**; RACT (Reactant or reagent)  
 (synthesis and deprotection-nitration of; in synthesis of TNFX,  
 (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

RN 333421-36-6 HCAPLUS  
 CN 1,5-Diazocine-3,3(2H)-diamine, N,N,N',N'-tetrafluoro-hexahydro-7,7-dinitro-  
 1,5-bis[(4-nitrophenyl)sulfonyl]- (9CI) (CA INDEX NAME)



RN 333421-37-7 HCAPLUS

CN 1,5-Diazocine-3,3(2H)-diamine, N,N,N',N'-tetrafluorohexahydro-7,7-dinitro-1,5-bis[(2-nitrophenyl)sulfonyl]- (9CI) (CA INDEX NAME)

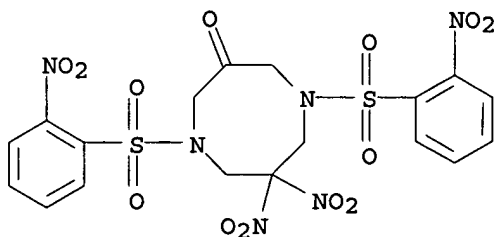


IT 333421-34-4P 333421-35-5P

RL: RCT (Reactant); SPN (Synthetic preparation); **PREP**  
**(Preparation)**; RACT (Reactant or reagent)  
 (synthesis and difluoramination of; in synthesis of TNFX,  
 (difluoramino)tetranitrodiazocine, as novel energetic heterocycle)

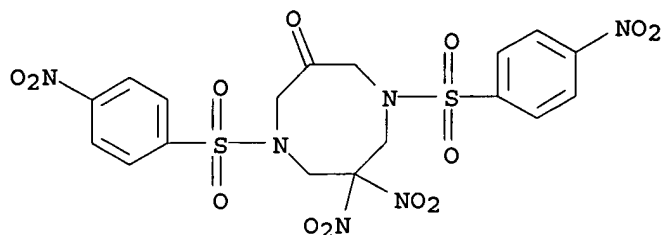
RN 333421-34-4 HCAPLUS

CN 1,5-Diazocin-3(2H)-one, hexahydro-7,7-dinitro-1,5-bis[(2-nitrophenyl)sulfonyl]- (9CI) (CA INDEX NAME)



RN 333421-35-5 HCAPLUS

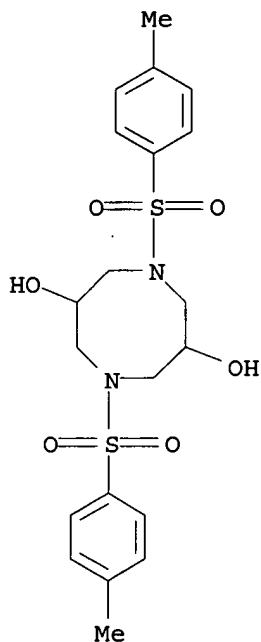
CN 1,5-Diazocin-3(2H)-one, hexahydro-7,7-dinitro-1,5-bis[(4-nitrophenyl)sulfonyl]- (9CI) (CA INDEX NAME)



RE.CNT 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 2 OF 2 HCAPLUS COPYRIGHT 2006 ACS on STN  
AN 1998:157886 HCAPLUS  
DN 128:140683  
TI Difluoramination of Heterocyclic Ketones: Control of Microbasicity  
AU Chapman, Robert D.; Welker, Mark F.; Kreutzberger, Charles B.  
CS Research and Technology Group (Code 4B2200D), Naval Air Warfare Center  
Weapons Division, China Lake, CA, 93555, USA  
SO Journal of Organic Chemistry (1998), 63(5), 1566-1570  
CODEN: JOCEAH; ISSN: 0022-3263  
PB American Chemical Society  
DT Journal  
LA English  
OS CASREACT 128:140683  
AB Difluoramination of a tetrahydro-1,5-diazocine-3,7(2H,6H)-dione to the  
corresponding 3,3,7,7-tetrakis(difluoramino)diazocine was achieved by a  
judicious choice of protecting group. Arenesulfonyl protecting groups for  
the diazocine nitrogens proved superior to acetyl during the slow  
disruption of the transannular bridge in 9-oxa-3,7-  
diazabicyclo[3.3.1]nonane intermediates by difluorosulfamic acid. While a  
1,5-ditosyl derivative failed to proceed beyond the product of addition of  
difluoramine to one ketone carbonyl, the use of 4-nitrobenzenesulfonyl as  
the protecting groups lowered the nitrogens' basicities below that of the  
oxygen site in the dione and intermediates, allowing the reaction to  
proceed to a gem-bis(difluoramino)diazocine product. A safer procedure  
for handling difluoramine is described.  
CC 28-23 (Heterocyclic Compounds (More Than One Hetero Atom))  
Section cross-reference(s): 50  
ST tetrahydrodiazocinedione difluoramination; diazocinedione tetrahydro  
difluoramination; difluoramination heterocyclic ketone; safety handling  
difluoramine  
IT Amination  
(difluoramination of tetrahydrodiazocinedione)  
IT Protective groups  
(effect of protecting groups in difluoramination of  
tetrahydrodiazocinedione)  
IT Safety  
(in handling difluoramine)  
IT 106-89-8, reactions 1510-31-2 6325-93-5 94683-14-4  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(difluoramination of tetrahydrodiazocinedione)  
IT 10405-27-3P, Difluoramine 160624-80-6P 202211-14-1P  
202211-16-3P 202211-17-4P 202211-18-5P 202211-19-6P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(difluoramination of tetrahydrodiazocinedione)

IT 202211-15-2P 202211-20-9P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(difluoramination of tetrahydrodiazocinedione)  
IT 94683-14-4  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(difluoramination of tetrahydrodiazocinedione)  
RN 94683-14-4 HCAPLUS  
CN 1,5-Diazocine-3,7-diol, octahydro-1,5-bis[(4-methylphenyl)sulfonyl]- (9CI)  
(CA INDEX NAME)

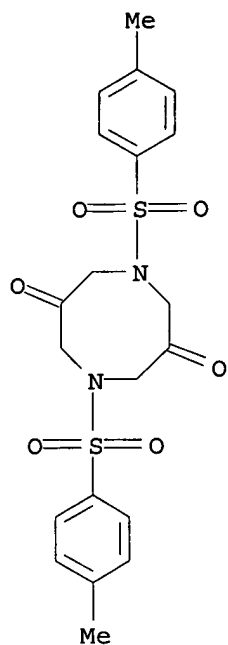


IT 10405-27-3P, Difluoramine 160624-80-6P  
202211-16-3P 202211-17-4P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(difluoramination of tetrahydrodiazocinedione)  
RN 10405-27-3 HCAPLUS  
CN Fluorimide (6CI, 8CI, 9CI) (CA INDEX NAME)

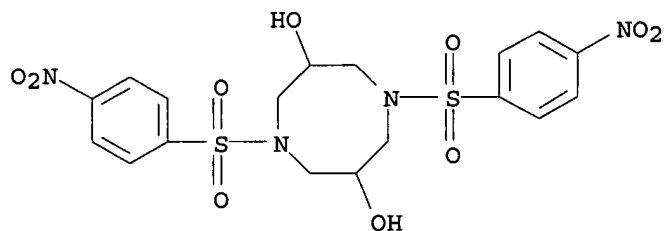
F-NH-F

RN 160624-80-6 HCAPLUS  
CN 1,5-Diazocine-3,7(2H,4H)-dione, tetrahydro-1,5-bis[(4-methylphenyl)sulfonyl]- (9CI) (CA INDEX NAME)

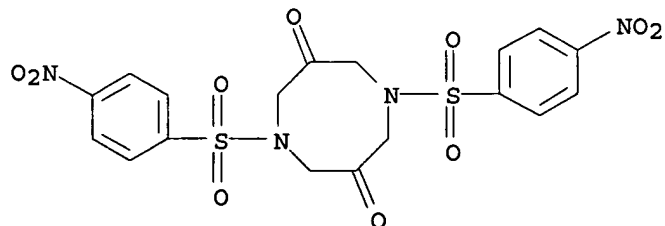




RN 202211-16-3 HCAPLUS  
 CN 1,5-Diazocine-3,7-diol, octahydro-1,5-bis[(4-nitrophenyl)sulfonyl] - (9CI)  
 (CA INDEX NAME)

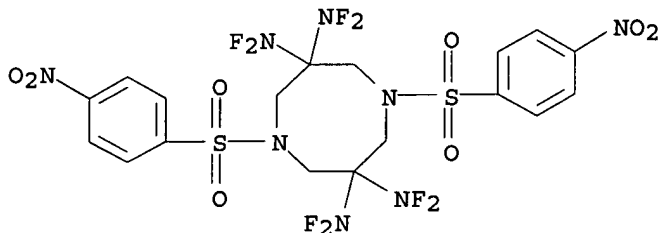


RN 202211-17-4 HCAPLUS  
 CN 1,5-Diazocine-3,7(2H,4H)-dione, tetrahydro-1,5-bis[(4-nitrophenyl)sulfonyl] - (9CI) (CA INDEX NAME)



IT 202211-20-9P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (difluoramination of tetrahydrodiazocinedione)  
 RN 202211-20-9 HCAPLUS

CN 1,5-Diazocine-3,3,7,7(2H,4H)-tetramine, N,N,N',N',N'',N'',N''',N''''-  
octafluorotetrahydro-1,5-bis[(4-nitrophenyl)sulfonyl]- (9CI) (CA INDEX  
NAME)



RE.CNT 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

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